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Methane sorption on bituminous coal – experiments on cuboid-shaped samples cut from primal coal lumps

Introduction

Environmental merits and potential economic aspects of enhanced coalbed methane recovery (ECBM) are the reasons for growth of the world wide interest in this field. The designation of correlations between sorption and induced swelling/shrinkage processes on coal and the understanding of the mechanisms that are the basis of these dependences are some of the most important aspects of ECBM recovery that still need to be researched. Hence during last couple of years it became perceptible that the number of studies in this field increased considerably. Basic problems that require analyzing are the changes induced by coal-gas (CH_4, CO_2) interactions in coal seams with regard to it as the specific gas reservoirs as well as the elaborating the analytical and numerical methods for description of these phenomena. Another reason for studying different aspects of methane sorption on coal are outbursts. Gases contained in hard coal seams, are present in coal beds of pressures above atmospheric pressure and releasing from coal during exploitation, due to decrease of external pressure (Ceglarska-Stefańska et al. 2008). Depending on the existing conditions, this process conducts with different rate and causes sorbed gas accumulated in the coal material to become free gas, which then flows through the strata's cracks and fissures towards the goaf areas, underground working, ventilation net work, and/or migrates to the surface. By high rates of gas releasing, gas and rock outbursts take place. The main causes of an

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ejection of gas and coal from the solid face are: high rock mass pressure at the coal bed which, often intensified by the mining activity, low strength of coal and high gas content of a coal bed.

Numerous considerations have been performed to estimate the relationships between the coal rank and its petrographic composition with regard to groups and single macerals, and the sorption and swelling/shrinkage properties of coal. The results of CH_4 sorption experiments on hard coals shown in papers (Mastalerz, Gluskoter, Rupp 2004) suggest that there is no clear correlation between coal reflectance, its petrographic composition and sorption capacity in CH_4-coal systems.

On the other hand, data presented in articles (Beamish, Gamson 1993; Chalmers, Bustin 2007; Lamberson, Bustin 1993; Levine 1993) indicate that increase of vitrinite content in coal petrographic composition correlates with a greater methane sorption capacity. This dependence reflects the large microporosity of this maceral group. Authors of the paper (Chalmers, Bustin 2007) also suggested that maceral composition has stronger influence on the sorption processes in the case of coals of higher rank, because of significant meso- and macroporosity of low rank coals, even within vitrinite group. Induced swelling/shrinkage of coal has been studied for some time (Czapliński 1966; Czapliński 1968; Ettinger 1974; Ceglarska-Stefańska, Czapliński 1977; Ceglarska-Stefańska, Czapliński 1979). The results of experiments generally indicate that coal as biporous system of dual transport and sorption nature, in which under high pressure of gas the microporous areas are being compressed and the macroporous ones are being widen, while swelling of microporous areas determines narrowing transport pores and the decrease of system permeability (Seewald, Klein 1986; Karacan, Mitchell 2003; Ceglarska-Stefańska, Zarębska 2006; Pan, Connel 2007). There are a number of articles that show experimental data indicating a linear correlation between coal expansion/contraction and the amount of sorbed gas, as in papers (Levine 1996; St. George, Barakat 2001; Chikatamarla, Cui, Bustin 2004; Robertson, Christiansen, 2005). However some tests results, like those presented in works (Ceglarska-Stefańska 1990; Pan, Connel 2007; Ceglarska-Stefańska et al. 2007, 2008) do not show linear dependence between sorption and induced strains.

In case of research, in which beside sorption processes the induced swelling/shrinkage behavior of coal is monitored, the size and shape of samples are of great importance, especially for choosing a method of dilatation measurements. There is also another important reason for applying block samples. As it needs to be stressed, the sieving process may result in partial enrichment or depletion of coal macerals in certain grain size fractions that would effect the sorption kinetics and gas capacity (Busch et al. 2004; Cygankiewicz, Dudzińska, Żyła 2009). Hence, the traditional sorption experiments on powders and grain samples are inadequate to modeling in situ conditions due to destruction of coal natural porosity and lack of stress from rock strata surrounding the seam (Karacan, Mitchell 2003).

The aim of this paper was to analyse the influence of coal rank and petrographic composition of hard coal on changes of the amount of sorbed methane and induced expansion/contraction of coal, with regard to cuboid-shaped samples.

1. Experimental

Cuboid –shaped samples of two coals, both high volatile bituminous C type according to ECE-UN In Seam Coal Classification and types 32.2 and 34.2 by PN-82/G-97002 were used. The coals were obtained, respectively, from KWK „Brzeszcze-Silesia”, seam 352 (coal B352) and from KWK „Budryk”, seam 401 (coal B1B) in the Upper Silesia Basin. For both specimens an elemental analysis, technical analysis and true density measurements were determined (Table 1). Samples B1B and B352 (20 × 20 × 40 mm) were cut from their primal lumps with a diamond saw, in the way that smaller sides (20 × 20 mm) were parallel and bigger sides (20 × 40 mm) were perpendicular to the bedding plane. Also the maceral compositions of the test material and vitrinite reflectance R° (sample B352) were obtained (Table 2). The petrographic analysis was carried out at the Faculty of Geology, Geophysics and Environmental Protection, AGH University of Science and Technology in Kraków, with the use of a mineralogical microscope and a reflectometer Zeiss-Opton).

The experiments conducted on samples B352 and B1B included investigation of the kinetics of methane accumulation and the kinetics of CH_4 sorption induced strains. Sorption and expansion/contraction measurements were taken using the apparatus and test procedures followed that described by (Majewska et al. 2009). The experimental set-up consisted of two

TABLE 1

Characteristics of the coal samples under study

TABELA 1

Charakterystyka materiału badawczego

	W^a [%]	A^a [%]	C^a [%]	C^{daf} [%]	H^a [%]	N^a [%]	S^a [%]	d_{true} 10^3[kg/m^3]
B352	2.3	4.09	77.53	82.82	4.56	1.43	0.22	1.339
B1B	0.7	1.8	88.6	90.9	5.66	1.41	0.63	1.291

TABLE 2

Petrographic compositions of the investigated coal samples

TABELA 2

Skład petrograficzny materiału badawczego

	Telinite	Telocollinite	Vitrodetrinite	Collodetrinite	Corpogelinite	Gelinite	**Total vitrinite**	Sporinite	Resinite	Cutinite	Liptodetrinite	**Total liptynite**	Fusinite	Semifusinite	Macrinite	Mikrinite	Funginite	Inertodetrinite	**Total inertinite**	**Mineral matter**	R° [%]
B352	2.8	33.7	0.3	9.6	0.3	1.7	**48.4**	7.8	0.5	0.0	1.6	**9.9**	15.8	11.2	1.1	0.9	0.2	8.4	**37.6**	**4.1**	0.77
B1B	0.2	15.5	0.0	48.0	0.0	0.6	**64.3**	3.7	0.4	1.0	0.0	**5.1**	10.7	5.2	2.0	0.2	5.4	7.1	**30.6**	**0.0**	–

individual units working together: (1) the gas sorption apparatus using volumetric method (pressure meter MKS BARATRON 722A with a measuring range of 0–4.0MPa and measurement accuracy ±0.001 MPa) and (2) the strain meter for measuring samples external dimensions changes induced by gas sorption and pressure (electrical resistance bridge, type SGM-1C81, constructed in Strata Mechanics Research Institute of The Polish Academy of Science, measuring range of linear strain ~4‰, measurement accuracy 0,001‰, with domestic production resistance-type paper strain gauges type RL120). The whole apparatus was thermo-stabilized, which enabled measurements to be taken at constant temperature of 298 K. Hence it was possible to monitor simultaneously the courses of methane accumulation and the development of the strains on monolithic coal samples.

The experimental procedure included the previous degassing of the sorption apparatus and the sample itself (vacuum 10^{-5} Pa) and immersion in a helium bath (10 kPa). For both systems, B352-CH_4 and B1B-CH_4, two kinetics of gas accumulation were monitored, determined by using the pressure progression technique for this two-stage sorption experiment. Therefore the concept stage-one describes the first step of the experiment, which included injection of methane to the degassed sample cell at fixed dosing pressure d_1 and investigating the changes of the amount of accumulated gas and sample external dimensions until the near-equilibrium state of sorption induced strains were reached at pressure p_1. While by stage-two is meant that after stage-one an injection of an additional portion of gas took place at pressure higher than p_1. The pressure in sample cell reached the d_2 value and then both accumulated gas volume changes and induced strain variations were further monitored. A symbol p_2 relates to the pressure value obtained in the ampoule at the end of the second step of the test. As a result of the experiments the kinetics of methane accumulation were determined as well as the kinetics of the induced strain perpendicular (ε_T) and parallel (ε_L) to the bedding plane, on the basis of which a kinetics of volumetric strain were calculated in accordance with the formula: $\varepsilon_V = \varepsilon_T + 2\ \varepsilon_L$.

2. Results and discussion

The results given as plots in Figure 1 and 2 show the curves of kinetics of methane accumulation in porous structure of cuboid-shaped coal samples, marked respectively as B352 and B1B and curves of kinetics of coal induced expansion/contraction. The axis of abscissa represents the time of contact between the sample and the sorbate (t [h]), the values on the left axis of ordinates show the swelling/shrinkage of coal (ε_V, ε_T, ε_L [‰]) and the right axis corresponds with the amount of gas accumulated in coal porous structure (V [cm^3STP/g]). The results of experiments on samples B352 and B1B have been cited in Table 3, Figure 3 and 4 illustrate the relationship between the expansion/contraction of coal, respectively B352 and B1B, and the amount of gas accumulated in their structures. On the horizontal axis the volume of methane in coal is shown (V [cm^3STP/g]) and the vertical axis represents the volumetric strain (ε_V [‰]).

TABLE 3

Changes of the amount of accumulated gas and induced strains of the coal samples under study

TABELA 3

Zmiany ilości zgromadzonego gazu i odkształceń badanych próbek

System B352-methane					
	Time [h]	Sorbed gas [cm^3STP/g]	Strain ε_L [‰]	Strain ε_T [‰]	Strain ε_V [‰]
Stage-one					
d_1 = 4.13 MPa	0	0.0	0.0	0.0	0.0
	48	3.10	1.16	1.38	3.76
p_1 = 3.59 MPa	570	4.67	1.96	1.97	6.03
Stage-two					
d_2 = 4.17 MPa	0	4.67	1.96	1.97	6.03
p_2 = 4.10 MPa	190	5.23	2.21	2.11	6.68
System B1B-methane					
	Time [h]	Sorbed gas [cm^3STP/g]	Strain ε_L [‰]	Strain ε_T [‰]	Strain ε_V [‰]
Stage-one					
d_1 = 2.67 MPa	0	0.0	0.0	0.0	0.0
	48	14.02	0.76	0.97	2.68
p_1 = 0.72 MPa	120	19.02	0.78	0.95	2.68
Stage-two					
d_2 = 4.13 MPa	0	19.02	0.78	0.95	2.68
	15	39.47	1.22	1.45	4.07
p_2 = 0.75 MPa	95	47.82	0.83	1.16	3.01

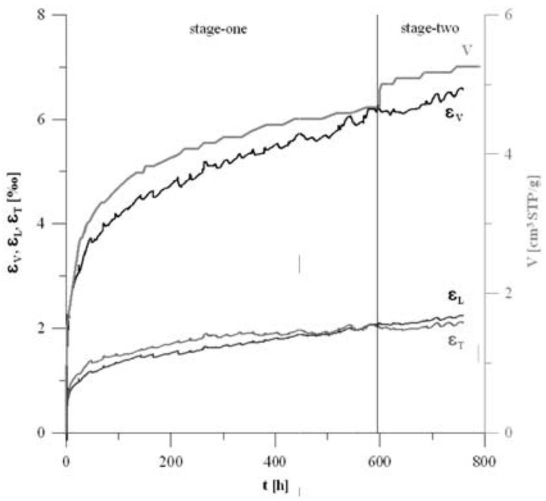

Fig. 1. Kinetics of methane accumulation and induced strains– sample B352

Rys. 1. Kinetyki akumulacji metanu i odkształceń węgla – próbka B352

Investigated systems coal B352-CH_4 and coal B1B-CH_4 show significant differences in both, the amounts of accumulated gas and samples induced external dimensions changes. The structure of coal B352 contained a few times smaller volume of methane than coal B1B. The induced strains show opposite trend. As the results clearly present expansion of sample B352 is greater than in the case of sample B1B and the courses of their strain kinetics show large dissimilarity (Fig. 1, 2).

Low gas capacity of sample B532 towards methane could be the result of low rank of this coal which relates with relatively low content of micro- and ultramicropores in the total

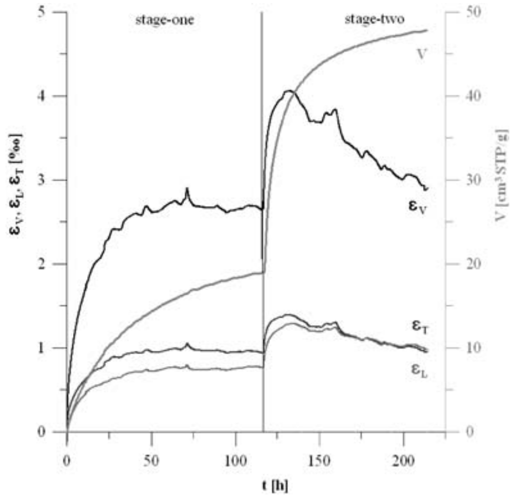

Fig. 2. Kinetics of methane accumulation – sample B1B

Rys. 2. Kinetyki akumulacji metanu i odkształceń węgla – próbka B1B

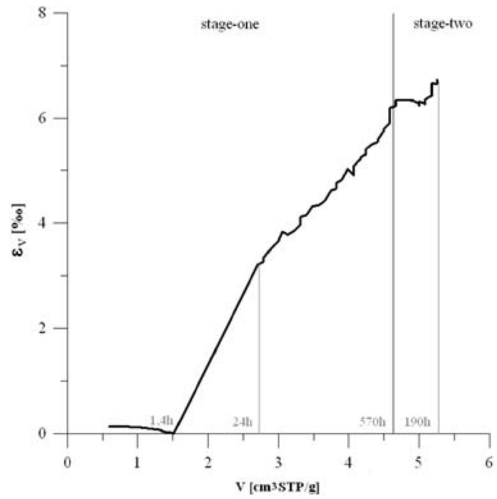

Fig. 3. Relationship between volumetric strain and accumulated gas volume – sample B352

Rys. 3. Zależność odkształceń objętościowych od ilości gromadzonego gazu – próbka B352

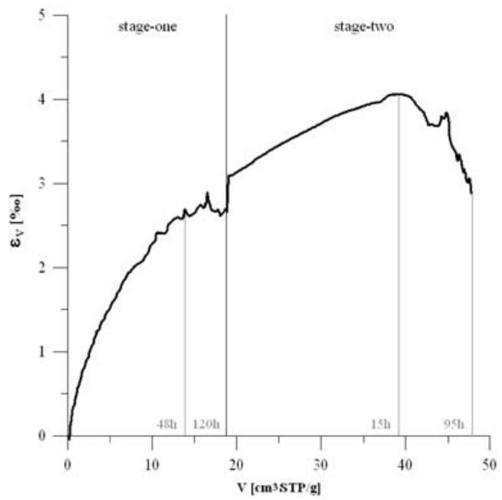

Fig. 4. Relationship between volumetric strain and accumulated gas volume – sample B1B

Rys. 4. Zależność odkształceń objętościowych od ilości gromadzonego gazu – próbka B1B

porosity of coal even in macerals of the vitrinite group (Chalmers, Bustin 2007). The presence of peripheral located functional groups is also significant. Acting as outlets they restrict the access to the pore structure to methane molecules. Another very important factor limiting the gas capacity of coal B352 is high inertinite and liptynite content in its petrographic composition, especially due to the presence of fusinite (15.8%) and semifusinite (11.2%). In the case of sample B352 this macerals are mostly not impregnated and have numerous empty spaces. Furthermore coal B352 contain only 48.6 % of vitrinite. Methane accumulation kinetics and adequate induced strain kinetics for coal B352 have a similar course. After 24 hours of the experiment the swelling of coal was roughly proportional to the volume of accumulated gas and relationship $\varepsilon_V = f(V)$ became linear (Fig. 3).

Taking into consideration the low rank of coal, the structure of B352 should be considered as highly disorder one. As a result of the lack of spatial orientation of the high-molecular compounds (crystalline-like domains) that form a three-dimensional network and significant amount of mobile molecular fraction (crosslinked and non-croslinked chaines of elastic compounds) the carbon matter of coal B352 while interacting with the CH_4 could become not only elastic but also capable of displacement of its structural elements in a wide range of sorption induced strains. However in this case there is no possibility to specify which of the factors: absorption process, adsorption process or free gas pressure in pores had the largest influence on the total volumetric strain in particular stages of the test. During the first several minutes of experiment the change of sample dimensions was inconsiderable. An analogous beginning of the experiment on coal-methane system (coal type 32.2 from KWK "Brzeszcze--Silesia") was presented in the work of Ceglarska-Stefańska (1990). The author assumed that course of early stage strain kinetics is related to some pores being inaccessible for CH_4 particles at the beginning and they are forced to overcome the energy required to widen these pores.

B1B is a coal of medium rank that has parameter C^{daf} about 85–90% (coking coals) that correlates with a minimum of porosity and a maximum of true density of hard coals. However the volume of methane accumulated in the structure of sample B1B was quite large. It could be possible considering relatively well developed microporous structure correlated with the presence of significant amount of vitrinite coal and an optimal system of transport pores with limited number of functional groups. Macerals of vitrinite group dominates in the petrographic composition of coal B1B (64.3%) but the amount of inertinite is also significant (total of 30.5 %). The course of accumulation kinetics for stage-one differs from the adequate plots for stage-two mainly with regard to the rates of gas uptake and deposition in the structure of coal B1B (Fig. 2). It was much higher in the case of stage-two. A conclusion can be drawn that increasing the dosing pressure of methane causes a higher rate of gas accumulation in the tested coal porous structure. Analogical findings were already presented in previous paper (Czerw, Ceglarska-Stefańska 2008).

It can be assumed that the structure of coal B1B can be described as compact, quite rigid and with poorly developed special orientation. Therefore in the case of this coal the easiness of external dimension changes induced by gas sorption and free gas uptake is limited as the movement abilities of its macromolecular fraction and molecular phase are rather blocked. Within the first 48 hours of experiment the rate of methane accumulation in coal B1B was greater than the swelling with reference to the hypothetical linear correlation of these two processes (Fig. 4). Then the increase of the sample dimensions in both directions, parallel and perpendicular to the bedding plane stopped. After that the size of the sample remained constant up till the end of stage-one, despite the progressing gas accumulation (Fig. 2, 4). A number of factors indicate that the lessening of tension in coal structure occurred which resulted in strain relaxation (with a constant deformation) and of course was linked with gas pressure decrease in coal specimen. On molecular level this relaxation could be related to weakening or breaking some of the chemical bonds between the elements of macromolecular network and displacement of the mobile molecular components that created new spaces for further gas accumulation throughout adsorption and absorption processes and as a free gas.

The induced strain kinetics for stage-two on sample B1B have much different course than those obtained during the first part of this test (Fig. 2). The methane dosing pressure was higher than the pressure previously used. Within the first 15 hours of the experiment the correspondence between the amount of up-taken gas and induced volumetric strain was a linear correlation. But after reaching a maximum value the relationship $\varepsilon_V = f(V)$ changed. The sample started to shrink and continued till the end of the experiment despite the gas accumulation in progress (Fig. 2, 4). It seams reasonable to state that using a higher dosing pressure had an effect on coal structural properties. The chemical bonds between the molecules had been broken and some molecules displaced. The deformation started to be non-reversible. Yet, after 15 hours, when the 97% of this stage gas capacity was reached, the rate of the changes in sorbate content in coal B1B porous structure became small enough to make the sorption induced changes in surface potential energy less than the work of free gas in pores and cleats (Pan, Connel 2007; Ceglarska-Stefańska, Czerw 2008). The increase of density and pressure of free gas became large enough to cause the compression of coal for which the sorption induced swelling was the counterpoise until the 15th hour of this experimental stage. As it was noticed before, in the case of coals with a medium content of element C, using a higher dosing pressure of sorbate increase the rate of methane accumulation in porous structure of coal and at the same time contraction is being observed in spite of the continuous gas uptake. In situ that can effect as the decrease of the hardness of coal solid and create the risk of gas and rock outburst.

Summary

Mining working environment is characterized by the presence of natural and technical hazards, to which people are exposed. Save work environment should be based on the knowledge referring to its components, like rock mass and gases, and their relationships that can dynamical change during operational activities. Therefore numerical modeling of the mine gas retention, accumulation and emission in and from coal seams with an adequate description of sorption induced coal swelling and shrinkage requires further research on relatively large solid samples of coal. In the case of monolithic specimens the natural heterogeneity of primal coal and its dual porosity as well as content of individual macerals and microlithotypes can be preserved.

Developing forecasts of coal-methane systems needs an individual approach, especially with regard to coals which differ in their ranks. This conclusion has its confirmation in experimental results published in present paper. This data indicate that the character of CH_4-coal interactions that goes together with the course of sorption processes and induced dimension changes in a large measure depend on the rank of coal. This parameter determines the coal structure on molecular level and its porosity, which follows results described by (Zarębska, Dudzińska 2008). Nevertheless the maceral composition is as important factor as the coal rank and has an influence on its interactions with gas sorbates.

The dependence of coal gas capacity and strain changes on the rank of coal and its petrographic composition may have significant consequences in the aspect of mine gas emissions and coalbed methane recovery. Coal mining methane and coalbed methane is a valuable fuel source. Therefore its recovery may be regarded as a beneficial and profitable element in the mine resources management.

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SORPCJA METANU NA PROSTOPADŁOŚCIENNYCH PRÓBKACH WĘGLA KAMIENNEGO

Słowa kluczowe

Węgiel kamienny, metan, kinetyki akumulacji, pęcznienie, skurcz, naprężenie

Streszczenie

Przeprowadzono badania sorpcji metanu na dwóch węglach, nisko- i średniouwęglonym (odpowiednio typ 32.2 i typ 34.2 wg PN-82/G-97002). Eksperymenty zrealizowano metodą objętościową na prostopadłościennych litych próbkach o wymiarach 20 × 20 × 40 mm wyciętych z bryły macierzystej. Równocześnie śledzono towarzyszące procesom sorpcyjnym zmiany zewnętrznych wymiarów próbek węgla. Dla każdego układu wyznaczono dwie kinetyki sorpcji, przy zastosowaniu metody progresji ciśnienia, oraz kinetyki odkształceń w kierunkach prostopadłym i równoległym do uławicenia węgla. Obliczono także kinetyki odkształceń objętościowych próbek.

Pozyskiwanie obok samego węgla również metanu z pokładów węglonośnych stanowi jedną z możliwości efektywnego wykorzystania i zagospodarowania obu tych surowców energetycznych. Jednocześnie, wysoka metanonośność złoża oznacza tym samym prawdopodobieństwo wystąpienia zagrożenia wyrzutem gazów i skał. Stąd określenie zależności pomiędzy sorpcją/desorpcją gazów a pęcznienie/kurczeniem się węgla w pokładzie na skutek przebiegu zjawisk sorpcyjnych oraz zrozumienie mechanizmów tych procesów stanowi nieodzowny element zarówno w kwestiach związanych z ECBM, jak i w zakresie bezpieczeństwa eksploatacji kopalń węgla. W pracy wykazano, że w przypadku węgli kamiennych stopień metamorfizmu oraz skład petrograficzny węgla należą do kluczowych czynników determinujących przebieg procesów sorpcyjnych i towarzyszących im odkształceń sorbentu. Podstawę rozważań stanowiły odmienne kinetyki sorpcji i skrajnie różne kinetyki odkształceń wyznaczone dla badanych układów.

Key words

Hard coal, methane, kinetics of accumulation, swelling, shrinkage, strain

Abstract

The paper reports the results of laboratory experiments concerning the accumulation of methane on two coals of different ranks, low- and medium-, (respectively coal type 32.2 and 34.2 by PN-82/G-97002). The tests were conducted on cuboid-shaped solid coal samples (20 × 20 × 40 mm) cut out of pieces of primal coals with a long axis parallel to the bedding plane and by applying the volumetric method. The changes of external dimensions which occurred during CH_4 accumulation (sorption induced strains) and the volume of up-taken gas were measured simultaneously. For every coal-gas system, two kinetics of gas accumulation were determined by applying the pressure progression method, as well as adequate kinetics of CH_4 induced strains parallel and perpendicular to the bedding plane. Volumetric strain kinetics were calculated.

The additional recovery of methane beside normal exploitation of coal seams pose as one of the possibilities of efficient management and use of both of those energy resources. On the other hand high concentration of methane in coal suggests the possibility of emerging of gas and coal outburst. Therefore the designation of correlations between sorption and induced swelling/shrinkage processes on coal and the understanding of the mechanisms that are the basis of these dependences are some of the most important aspects of ECBM recovery and mining operational safety. The presented study has demonstrated that in case of hard coals, the rank and the petrographic composition are two of the key factors that influence the course of sorption processes and the methane induced swelling/shrinkage of coals. The above conclusion was based on the strong differences in courses of acquired strain and gas capacity kinetic curves.